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EVALUATION OF THE USE OF EMULSIFIERS TO IMPROVE THE PRODUCTION OF HEAVY OIL

(PART I)

Final Report

By

M. Sarbar
Texaco Canada Resources

F.M. Mourits and L. Kurucz
Saskatchewan Research Council
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SUMMARY

This study was undertaken to evaluate the technical feasibility of emulsification of Frog Lake and Lone Rock wellhead crudes. The creation of oil-in-water (o/w) emulsions downhole may significantly improve pumping and production efficiencies.

A range of eight o/w emulsifiers (Petrofirm HO Flow, Tretolite CF-4W and PD-57, BASF Pluronic P-85, SRC-K1, SRC-K8, Pfizer F-2, and Pfizer F-10) was evaluated at concentrations varying from 250-2000 ppm. All three crudes could be emulsified; the ease of emulsification, the stability and the viscosity of the o/w emulsions created was determined by the combination of emulsifier type and concentration.

The emulsification process left some residual water in the low-water-content Frog Lake crudes, which could not be removed by the demulsifier currently in use in the field (Champion 888X). Using the high-water-content Lone Rock crude for emulsification, only the SRC-K1 emulsifier was compatible with Champion 888X and did not affect the efficacy of this demulsifier.

Experiments with brines of reduced salinity showed that o/w emulsion stability increased with lower salt concentrations. On the other hand, o/w emulsion stability was adversely affected by the presence of sand. Only the most stable emulsions (prepared with Pfizer Flothin F-2) survived the presence of 15 vol % sand-in-oil, but sand dropout was rapid during the first 30 minutes. At lower sand concentrations (5 and 2 vol % sand-in-oil), other demulsifiers could also be used and sand dropout slowed down.

The above lab-bench results could be further elucidated by scaled pipeline loop and/or wellbore model tests.

Preferred downhole injection methods involve continuous injection (i) down the annulus, (ii) through hollow sucker rods or (iii) through spaghetti strings. Because of low capital costs, the first method is being used most frequently. Low fluid levels are desirable, if a short response time is preferred.

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I. INTRODUCTION

In the production of heavy oil, light hydrocarbons (diluent) have been used to reduce the viscosity and to improve the productivity of wells that have high fluid levels. The diluent is commonly injected downhole to decrease the viscosity of the heavy crude in the tubing and flow lines and to improve pumping efficiency. Occasionally, heat stimulation was used to achieve this reduced viscosity. An alternative method of improving production of heavy oils is to create downhole water-continuous emulsions, which will decrease the viscosity significantly and improve pumping and production efficiencies. This process will increase pumping speed, reduce power consumption and may make the check valves in rod pumps water-wet. The latter may minimize sand adhesion to the check valves, which in turn will result in a more consistent daily production for progressive cavity pumps. The process will also reduce the viscous drag on the rotating shafts of screw pumps, allowing the rotational speed of these pumps to be increased, which again will result in improved pumping efficiency. An important application of downhole emulsification may be found in fireflood operations, where frequently occurring highly viscous water-in-oil (w/o) emulsions could significantly reduce pumping efficiencies. The downhole emulsification process is the subject of the following study.

2. OBJECTIVES

The study described in this report was undertaken to evaluate and select wellbore emulsifiers, which can be used to help improve production of heavy oils. The following was to be accomplished:

(a) Evaluation of the degree of oil-in-water (o/w) emulsification and of the stability of the emulsions formed in the presence of various emulsifiers at different concentrations.

(b) Determination of the amount of viscosity reduction achieved by forming o/w emulsions.

(c) Assessment of the compatibility of the emulsifiers screened with the demulsifiers used in the field for oil treatment.

(d) Examination of the effect of salinity on o/w emulsion stability.

(e) Examination of the effect of sand on o/w emulsion stability and determination of sand dropout rates.

(f) Survey of current methods for emulsifier injection and identification of the best injection strategy.

(g) Identify the surface facilities required to perform the field test.

3. EXPERIMENTAL

3.1 Emulsion Preparation

Amounts varying from 0.03 to 0.2 g emulsifier were added to 33 mL brine. This solution was mixed for a few minutes to dissolve the emulsifier using a Cole-Parmer impeller-type variable speed mixer. Sixty-seven millilitre of the wellhead emulsion to be emulsified was then slowly added while mixing. After all the emulsion had been added, it was stirred for two minutes at low speed, followed by five minutes at medium-high speed. Assuming an equal solubility of the emulsifier in both oil and water phase, the overall concentration would vary from 250 to 2000 ppm. Immediately after the stirring was completed, the viscosity of the fresh o/w emulsion was determined using a Brookfield HBTDCP viscometer, equipped with cone and plate spindle.

After mixing, the o/w emulsion was placed into a 100-mL bottle and its stability determined by measuring the time it took for the water to break out and the original water-in-oil emulsion to reappear. The water dropout was determined at 15 min, 30 min, 1 h, 2 h, 3 h, 4 h and 24 h at ambient temperature. In selected cases the BS&W content of the oil layer was also determined after 24 h at ambient temperature (see Section 3.3).

The effect of sand on o/w emulsion stability was studied by using artificial wellhead emulsions with increased sand concentrations. These emulsions were prepared by mixing various amounts of sand in the wellhead emulsions provided. The same o/w emulsification procedures as described for the 100-mL bottle emulsification tests were then followed, except that 500-mL samples were prepared (for increased accuracy) and 1000-mL graduated cylinders were used for measuring the water and sand dropout rates. Also, the oil and emulsifier solution were mixed longer, i.e. 15 min at low speed, followed by 1 min at high speed. Water and sand dropout were determined at 0.5 or 1 h, 24 h and 6 d at room temperature. As a further check on sand retention by the oil, BS&W values of the oil layer were determined

at 24 h and 6 d in some cases.

The following quantities of original wellhead emulsion, sand, brine and emulsifier were mixed.

15 vol % sand-in-oil: 80 g sand (50 mL)
285 g oil
165 g brine
0.5 g emulsifier (1000 ppm)

5 vol % sand-in-oil: 27 g sand (17 mL)
318 g oil
165 g brine
0.5 g emulsifier (1000 ppm)

2 vol % sand-in-oil: 11 g sand (7 mL)
328 g oil
165 g brine
0.5 g emulsifier (1000 ppm)

3.2 Compatibility Studies

Compatibility studies were performed to ensure that the surfactants used for producing the o/w emulsions were compatible with the demulsifiers used to treat the oil. In principle, at least two bottle tests are required. The first involves preparing the o/w emulsion with an emulsifier as outlined above and allowing it to settle out. The free water is then removed and discarded, and a bottle test (Section 3.3) on the remaining w/o emulsion is performed with the required amount of demulsifier. The second bottle test (a blank test) is performed on the original w/o emulsion without the o/w emulsification step, using the same demulsifier concentration as in the first bottle. If there is any incompatibility between the emulsifier and the demulsifier, the final BS&W content of the converted and

treated emulsion would be somewhat higher than that of the original, treated emulsion.

A complication arose when testing the two Frog Lake wellhead samples. Because of their very low water content, incompatibilities, if any, could not be adequately detected. This problem was overcome by artificially raising the water content of the two wellhead emulsions. This was achieved by mixing 75 vol % wellhead emulsion and 25 vol % brine, resulting in w/o emulsions of 25-27 vol % BS&W. The latter emulsions were then used as the starting material for the compatibility tests.

3.3 Determination of Basic Sediment and Water

The amount of water and solids present in a water-in-oil emulsion is determined by the following procedure, historically called BS&W (basic sediment and water) determination.

A sample of the emulsion is diluted 50-50 with toluene in a 15-mL centrifuge tube. It is then centrifuged for 15-20 minutes. After centrifuging, the emulsion separates in most cases into three layers. The top layer is the clean oil and toluene, while the middle layer separates out as a high-water content emulsion, which is usually brown in colour. The bottom layer consists of free water and solids, if they are present. The total volume of free water (FW) and emulsion (E) is noted, along with a breakdown of each. Two to three drops of a batch-treating demulsifier (knock-out drops) are then added to the sample and mixed. It is then centrifuged again for approximately 15 minutes. The presence of the knock-out drops breaks up the emulsion layer, giving the total water (TW) and solids (S) content of the original emulsion.

Bottle Test

The bottle test is a widely used method to test the effectiveness of a

chemical demulsifier, an emulsion destabilizer or any combination thereof to break a water-in-oil emulsion.

A 100-mL sample of emulsion is placed in a 150-mL prescription bottle. A demulsifier or emulsion destabilizer at the required concentration is added to the bottle and the sample is thoroughly mixed by shaking the bottle. If the emulsion is too viscous for shaking, it can be preheated before the addition of the chemicals in order to lower the viscosity.

After mixing, the bottle is placed in a heated bath and allowed to sit. Bath temperatures will depend on the type of emulsion and its stability. Most bottle tests are conducted in the range of 60-90°C. It should be noted that for comparison a blank bottle test should also be run. In a blank test no demulsification or destabilizing chemicals are added to the bottle. The amount of water settling out in the bottle is noted, usually at half hour intervals or shorter if the water dropout rate is rapid.

When the bottle has reached a state where it appears little or no more water is settling out, the bottle is given four or five gentle mixes or rolls to aid in settling out any larger water drops, which may be hung up in the oil layer. It is then placed back in the bath until it has reached a point where no further water is settling out. A BS&W determination of the oil layer is subsequently carried out. The sample for the BS&W determination is taken approximately 0.6 to 1.2 cm above the oil-water interface by a syringe with a long needle.

From the BS&W values the effectiveness of the demulsifier or emulsion destabilizer can be assessed. The presence at the oil-water interface of an emulsion pad, if it exists, should be noted since ideally the interface should be a sharp, distinct layer between the water and oil.

4. RESULTS AND DISCUSSION

4.1 Crude Oils Studied

Characteristics of the three crude oils studied are provided in Table 1. The viscosity of the wellhead emulsions varied from about 24,000 mPa.s for the heaviest Frog Lake crude to 3800 mPa.s for the lighter Lone Rock crude. The water content of the Frog Lake crudes was very low to nil, whereas the Lone Rock crude contained 54% water. The sand content of the two Frog Lake crudes was relatively small (0.3-0.4%), the Lone Rock crude did not contain measurable quantities of sand. The TDS (total dissolved solids) contents of the three brines produced by these wells fell in a narrow range (70,000-80,000 ppm by weight).

4.2 O/W Emulsification Characteristics of Texaco Frog Lake Wellhead Crude (8-13-57-2 W4M)

The above wellhead crude was emulsified using eight different emulsifiers at concentrations varying from 2000 to 250 ppm (Table 2). This series of emulsifiers spanned a wide range.

Petrofirm's HO Flow formed one extreme; it was generally poorly water-wetting and produced fairly unstable emulsions consisting of visually large oil drops. The instability of the o/w emulsions prepared with HO Flow was evident from the rapid water dropout. The instability increased as the emulsifier concentration was decreased from the initial 2000 ppm value. At 500 ppm the o/w emulsion became marginal and at 250 ppm reversal to an w/o emulsion occurred.

The two Tretolite emulsifiers (CF-4W and PD-57) behaved very similar to HO Flow. In all three cases broken-out water was clean, but large oil drops remained attached to the glass walls whenever HO Flow was used.

The two SRC-K emulsifiers formed a middle group. They were moderately water-wetting and produced stable o/w emulsions with small oil droplets, especially at higher concentrations (1000-2000 ppm). O/W emulsions produced by SRC-K1 were less stable than those produced by SRC-K8 at the same concentration. Below 1000 ppm, SRC-K1 emulsions became unstable and reversed to w/o emulsions. Broken-out water was generally oily.

The BASF Pluronic P-85 formed the transition to the other extreme, where the two Pfizer Flothin emulsifiers were found. These three surfactants were fairly good (P-85) to very good water-wetting (Flothin) and produced excellent, stable emulsions with small oil drops at concentrations above 1000 ppm. Below 1000 ppm the emulsions became rapidly less stable or reversed to w/o emulsions (Flothin F-10, 500 ppm). In all three cases broken-out water was very oily.

The above observations closely paralleled the measured viscosities of the freshly-prepared emulsions (Table 5). Unstable emulsions were characterized by a high viscosity, due to the large size of the suspended oil drops. The original oil viscosity of 24,019 mPa.s was reduced to less than 1000 mPa.s by SRC-K1 (2000 ppm), Pluronic P-85 (1000 ppm) and Flothin F-2 (1000 ppm).

The BS&W content of the oil layer after settling for 24 h was determined in a few selected cases. Water content ranged from 8 vol % (HO Flow at 1000 ppm and SRC-K1 at 1000 ppm) to 14 vol % (Flothin F-2 at 1000 ppm).

Because of the similarities of the emulsifiers within the above three groups, one representative member of each group was chosen to be used in all following tests. The selected emulsifiers were HO Flow, SRC-K1 and Flothin F-2.

4.3 O/W Emulsification Characteristics of Texaco Frog Lake Wellhead Crude (14-6-57-1 W4M)

The characteristics of the O/W emulsions prepared with HO Flow, SRC-K1 and Flothin F02 generally showed the same features as those described in Section 4.2. There was, however, a trend towards lower stability with this wellhead crude. This can be seen from the increased water dropout rate for SRC-K1 at 2000 ppm and Flothin F-2 at 1000 ppm. Thus, it appears emulsifier concentrations would have to be increased to achieve comparable stabilities as obtained with Frog Lake crude 8-13.

The viscosity data of Table 6 confirm that despite the lower viscosity of the wellhead crude (12,664 mPa.s) and, consequently, of the o/w emulsions prepared, low viscosities (i.e. less than 1000 mPa.s) were achieved at generally higher emulsifier ppm levels.

4.4 O/W Emulsification Characteristics of Texaco Lone Rock Wellhead Crude (B5-14-47-27 W3M)

The Lone Rock wellhead crude was different from the two Frog Lake crudes because of its relatively low viscosity and its high (54%) water content. The latter further reduced the stability of the o/w emulsions as compared with the Frog Lake 14-6 data. SRC-K1 exhibited a unique feature, observed at numerous other occasions, namely the capability to cause the emulsified water in the wellhead crude to drop out. As a consequence, water dropout volumes with SRC-K1 were substantially higher than the amount of water used for emulsification (33 vol %), while the BS&W content of the oil layer after 24 h was lower.

As can be seen from Table 7, the viscosities of the o/w emulsions fell in a much narrower range. This, of course, is due to the relatively low viscosity of the wellhead crude. Remarkably, SRC-K1 achieved the lowest viscosities at both the 1000 and 2000 ppm levels. This is

attributed to the presence of the additional water broken out by SRC-K1.

4.5 Compatibility Studies

Compatibility tests as described in Section 3.2 were initially performed on the as-received wellhead emulsions. Because of the low water content of the two Frog Lake crudes, the final BS&W results for these oils as shown in Table 8, although higher than before emulsification, are somewhat ambiguous. For this reason, artificial wellhead samples with increased water content were prepared by mixing 67 vol % Frog Lake crudes with 33 vol % brine. These artificial crudes were subsequently emulsified (data in Table 9) and the resultant oils (after 24 h) subjected to standard demulsification bottle tests (Table 10).

The final BS&W results for the 8-13 Frog Lake crude show that all three demulsifiers were compatible with the Champion 888X demulsifier and did not cause any increase in BS&W content. HO Flow achieved a slightly lower BS&W content than the other chemicals or 888X alone. The results for Frog Lake 14-6 also show that the three demulsifiers were compatible with 888X, but more variation is observed in this case. SRC-K1 achieved a substantially lower BS&W than the other chemicals or 888X alone. In conclusion, for these Frog Lake crudes with increased BS&W, no incompatibilities could be detected.

The situation was quite different with the 54%-BS&W Lone Rock crude (Table 8). Regular demulsification with Champion 888X reduced the water content to 0.8%. Only emulsification with SRC-K1 could match this figure (1.0% BS&W). HO Flow and, especially, Flothin F-2 with its final BS&W figure of 18% were quite incompatible.

4.6 Salinity Effect on O/W Emulsion Stabilities

O/W emulsions were prepared with the three selected emulsifiers (at concentrations of 1000, 500 and 250 ppm), using Frog Lake 8-13 wellhead

crude and 8-13 brine at three concentrations (undiluted, ten times diluted and 100 times diluted). As expected (see Table 11), a trend toward increased o/w emulsion stability at higher brine dilution factors emerged. This trend was barely noticeable with HO Flow, but it was quite evident with SRC-K1 and Flothin F-2, especially at lower concentrations. At concentrations of 500-1000 ppm for SRC-K1 and 250-500 ppm for Flothin F-2, changing from undiluted to diluted brine (10x) caused a significant jump in o/w emulsion stability.

4.7 Sand Toleration Studies

Experiments were conducted to determine the effect of o/w emulsification on sand dropout rates. Due to the water-wetting nature of o/w emulsifiers, already water-wet sand particles are not expected to remain suspended in the oil phase, but to drop out. This could lead to potential problems with sanding of downhole pumps.

Table 12 shows the results of o/w emulsification of Frog Lake 8-13 crude to which 15 vol % sand had been added. Since emulsification involved using 67 vol % crude and 33 vol % brine, a 10-vol % sand concentration on an overall basis was obtained. In order to compare sand dropout rates, two blanks were prepared. Blank I was the wellhead crude itself with the added sand. No sand dropped out after 24 and 48 h at a slightly elevated temperature (30°C). Blank II underwent the o/w mixing procedure except that no emulsifier chemicals were added. The water was absorbed to form a w/o emulsion. After 48 h at 30°C, only 2 vol % white sand had settled out. The remainder was dispersed throughout the w/o emulsion in large pockets of clean sand.

The o/w emulsification results using emulsifiers show that the sand had a strongly destabilizing effect on the o/w emulsions, causing these emulsions to reverse to w/o emulsions. Only o/w emulsions prepared with the emulsifier that had produced the most stable emulsions in the previous

experiments (Flothin F-2 at 1000 ppm or higher) survived, but water and sand dropout were rapid. Essentially all of the sand added had dropped out after 30 min.

Using the same Frog Lake crude (8-13), further experiments were conducted with lower sand concentrations, i.e. 5 vol % sand-in-oil and 2 vol % sand-in-oil. The 5 vol % sand results (Table 13) show that suitable o/w emulsions could be prepared with SRC-K1 and Flothin F-2. Essentially no sand dropout could be observed after one hour, while the amount of dropped-out sand after 24 h and 6 days was very similar for the two o/w emulsions (as well as for the w/o emulsion formed with HO-Flow). Because of the difficulty determining such small quantities in the large 1000-mL graduated cylinders, a further check was made by determining the amount of sand left in the oil, i.e. by BS&W determinations. The BS&W data (Table 15) indicate that the amount of sand after 6 days was similar in all three oils. The high water content in the Flothin F-2 case indicates the presence of an o/w emulsion, even after 6 days.

A further reduction of the amount of sand mixed in the oil (2 vol %, see Table 14) made it possible for HO Flow to form an o/w emulsion, albeit only a fair one. No sand settled out in one hour in all three cases and only traces after 24 h. BS&W data (Table 15) show that after 24 h and 6 days the emulsions prepared with HO Flow and SRC-K1 retained most of the sand. Again, the higher water content of the Flothin F-2 samples supports the presence of o/w emulsions. This was also evident from the ease with which the oil layer could be sucked up for the BS&W determinations.

The results from sand dropout experiments with the Frog Lake 14-6 wellhead crude containing 15 vol % sand-in-oil (Table 16) parallel those with the 8-13 crude: only Flothin F-2 produced an o/w emulsion. As was the case with the regular (no sand added) wellhead crude experiments, the 14-6 crude yielded a less stable o/w emulsion at the same emulsifier concentration. Sand dropout rates and volumes were very similar, however.

Lone Rock B5-15 wellhead crude containing 15 vol % sand could be emulsified with both SRC-K1 and Flothin F-2 (Table 17). As was the case with the other two crudes, sand dropout was fairly rapid the first 30 min but then leveled off. Although Flothin F-2 first appeared to drop out more sand, the sand volumes for both emulsifiers were comparable after 24 h and amounted to only 50% of the total quantities of sand added. Whereas increased water dropout for high-BS&W crudes is common when SRC-K1 is used as emulsifier (possibly the reason why SRC-K1 produced a stable emulsion in this case), this phenomenon has thus far not been observed for other emulsifiers (see discussion in Section 4.4 for the Lone Rock crude). The presence of sand apparently changed this, as the water dropout in the case of Flothin F-2 was considerably higher than the 33 vol % added.

5. FIELD TEST

5.1 Surface Facility & Injection Strategies

The following approaches to downhole emulsification have been tried or are being proposed for field application:

1) injection down the annulus
2) injection through hollow sucker rods
3) injection through a small tubing (spaghetti string).

Injection can be carried out batch-wise and continuously. Adding the chemical batch-wise through the annulus has probably been carried out most frequently, as it is a "quick-and-dirty" way of trying out the concept. The results have been inconsistent (no control over injection rate) and in some cases caused some further problems.

Continuous injection through a hollow sucker rod or a spaghetti string is probably the best approach, but requires capital investments. Norcen used hollow sucker rods for their tests at the Bodo fireflood project, but these rods were already in place.

Continuous injection down the annulus could be a good compromise and requires minimal capital expenditures. However, in order to ensure that the chemicals quickly find their way to the pump, fluid levels in the annulus should be relatively low. High fluid levels do not preclude emulsification down the annulus, but it will take longer to saturate the fluids in the annulus with the emulsifier. This method of downhole emulsification is currently followed at Murphy's Eyehill and the Lindberg fields.

For wells with a higher fluid level, the surfactant could be diluted to a 1:10 ratio with water. This will ensure a faster delivery of the emulsifying chemical down to the production pump where it is required for the emulsification process. The surface facility required to perform downhole emulsification is shown in Figure 1. For wells producing over 20% water only the facilities shown on the upper half of Figure 1 are required to inject the chemical for downhole emulsification. For wells with a higher fluid level, or a lower water cut (less than 20%), the injection of water with the emulsifying chemical is also required to facilitate the emulsification process. Therefore, the whole injection system including chemical injection, as well as water injection facilities as is shown in Figure 1, are required.

5.2 Recommendation for Field Application

1. Continuously inject Flothin F-2 at a 500-1,000 ppm concentration (based on the total fluid production) down the annulus (see Figure 1 for the injection facility).
2. If the water cut in the produced fluid is less than 20%, then inject produced water into the annulus to increase the total water production to the 20% level.
3. When the sand content of the produced fluid is more than 5%, the downhole emulsification technique should be applied with a lot of precaution to avoid possible sand-off of the well.
4. If the production fluid contains less than 20% water cut and water injection is required into the annulus, then in order to minimize the chemical consumption, it is desirable to re-use the produced water that contains chemicals after the separation of oil and water at the surface.

6. CONCLUSIONS

1. The three wellhead crudes studied could be emulsified into o/w emulsions using a variety of emulsifiers.

2. Ease of emulsification, stability and viscosity of the o/w emulsions can be tailored by selecting suitable emulsifier/concentration combinations. The least stable, most viscous emulsions were produced with HO Flow, the most stable, least viscous emulsions with Flothin F-2.

3. Using Frog Lake crudes with increased water content, three emulsifiers tested (HO Flow, SRC-K1 and Flothin F-2) did not show any incompatibility with the treating demulsifier Champion 888X. Using the as-received low-water-content Frog lake crudes, the emulsification process resulted in a residual water content higher than before emulsification. Using the Lone Rock wellhead crude, only SRC-K1 was compatible with the Champion 888X demulsifier.

4. Reduced salinity of the brine used for emulsification leads to more stable o/w emulsions.

5. Sand destabilizes o/w emulsions. Using low-water-content Frog Lake crudes with high sand concentrations (15 vol % sand-in-oil), only Flothin F-2 was capable of producing o/w emulsions. However, sand dropout was rapid and most sand was released during the first 30 minutes. At lower sand concentrations (2-5 vol % sand-in-oil) SRC-K1 and HO Flow could also be used. Sand dropout at these concentrations slowed down significantly. Using a high-water-content Lone Rock crude with a high sand content, both SRC-K1 and Flothin F-2 produced suitable emulsions. Again, most sand dropped out during the first 30 min, but the overall amount was less than with the Frog Lake crudes.

6. Preferred downhole injection methods include continuous injection (a) down the annulus, (b) through hollow sucker rods, and (c) through a spaghetti string. The first approach requires little capital expenditures, but a low fluid level in the annulus would be desirable if a quick response is desired.

Table 1. Crude Oils Studied

Well	Formation	Formation Temperature (°C)	Crude Oil Viscosity (mPa.s)	BS&W (vol %)				TDS of Brine (ppm)
				FW	E	TW	S	
Frog Lake								
8-13-57-2 W4M	McLaren	22-23°C	24,019	0	0.3	0	0.4	71,354
14-6-57-1 W4M	Waseca	22-23°C	12,664	0	4.2	2.6	0.3	77,226
Lone Rock								
B5-14-47-27 W3M		20°C	3,810	36	20	54	0	82,094

Table 2. O/W Emulsification Characteristics of Texaco Frog Lake (McLaren) Wellhead Crude (8-13-57-2 W4M)

Emulsifier (ppm)	Water Dropout (Vol %) at Room Temperature							BS&W (Vol %) After 24 h		
	5 min	30 min	1 h	2 h	3 h	4 h	24 h	FW	E	TW
HO Flow[a]										
2000	-	18	20	20	20	20	20			
1000	-	20	20	20	25	25	25	7	1	8
500	25	30	30	30	30	30	30			
250	W/O									
HO Flow (Texaco)										
1000	5	20	20	-	-	20	-			
Tretolite CF-4W[b]										
2000	-	20	20	20	22	22	22			
1000	-	20	25	25	25	25	25			
500	20	25	30	30	30	30	30			
Tretolite PD-57[b]										
2000	-	15	19	20	20	20	20			
1000	15	20	22	22	22	22	22			
500	20	25	25	25	25	25	25			
Pluronic P-85[c]										
2000	-	3	8	10	15	15	20			
1000	-	5	8	15	-	-	22			
500	-	20	22	25	-	-	25			

(continued)

[a] Manufactured by Petroferm

[b] Manufactured by Petrolite

[c] Manufactured by BASF

Table 2. O/W Emulsification Characteristics of Texaco Frog Lake (McLaren) Wellhead Crude (8-13-57-2 W4M)--Continued

Emulsifier (ppm)	Water Dropout (Vol %) at Room Temperature							BS&W (Vol %) after 24 h		
	5 min	30 min	1 h	2 h	3 h	4 h	24 h	FW	E	TW
SRC-K1[d]										
2000	-	5	15	18	20	20	20			
1000	-	20	25	25	25	25	25	8	14	8
500	W/O									
250	W/O									
SRC-K8[d]										
2000	-	3	5	10	15	15	20			
1000	-	22	25	25	30	30	30			
500	-	25	30	30	30	30	30			
Flothin F-2[e]										
2000	-	3	3	5	5	8	18			
1000	-	3	3	8	15	15	18	13	15	14
750	-	15	20	22	25	25	25			
500	20	22	25	25	25	25	25			
Flothin F-10[e]										
2000	-	5	7	-	15	-	20			
1000	-	3	5	10	15	15	20			
500	W/O									

(continued)

[d] Manufactured by Hart

[e] Manufactured by Pfizer

Table 2. O/W Emulsification Characteristics of Texaco Frog Lake (McLaren) Wellhead Crude (8-13-57-2 W4M)--Continued

Emulsifier (ppm)	Comments on Emulsion Preparation
HO Flow	2000 ppm: Complete dispersion at 20-40 s. Good emulsion but relatively large oil drops.
	1000 ppm: Good emulsification after 2 min, with approx. same drop size as with 2000 ppm.
	500 ppm: Dispersion after 40-60 s, but marginal emulsion. Visually very large oil drops.
	250 ppm: Reversed to w/o emulsion at 6 min.
	HO Flow is poorly water wetting. The broken-out water phase is clear, but large oil drops are stuck to the glass container.
Tretolite CF-4W Tretolite PD-57	2000-500 ppm: Good emulsions after high-speed stirring. Both emulsifiers are poorly water-wetting. Broken-out water is clear.
SRC-K1	2000 ppm: Complete dispersion after 10-25 s, good to excellent emulsion. Visually small drops.
	1000 ppm: Good emulsion. Visually small drops.
	500 ppm: Dispersion complete at 15-30 s. Poor emulsion. Although the drop size was initially relatively small, it increased rapidly at 4 min. At 7 min the emulsion consisted of large globs and started to reverse to a w/o emulsion.
	250 ppm: Reversed to w/o emulsion after 2 min.
SRC-K8	2000-1000 ppm: Good to excellent emulsions.
	500 ppm: Marginal emulsion but no reversal to w/o.
	SRC-K1 and SRC-K8 are moderately water-wetting. Broken-out water is oily.
Pluronic P-85	2000-500 ppm: Good to excellent emulsions, but broken-out water is very oily. Fairly good water-wetting.
Flothin F-2	2000-750 ppm: Complete dispersion at 10-45 s. Excellent emulsions of visually small oil drops.
	500 ppm: Complete dispersion at 15-30 s. Marginal emulsion, increased oil drop size.
Flothin F-10	2000-1000 ppm: Excellent emulsions.
	500 ppm: Reversed to w/o emulsion after 3 min.
	Both Flothin emulsifiers are very good water-wetting. Broken-out water is very oily.

*Stirring time: 2 min low speed, followed by 5 min medium-high speed.

Table 3. O/W Emulsification Characteristics of Texaco Frog Lake (Waseca) Wellhead Crude (14-6-57-1 W4M)

Emulsifier (ppm)	Water Dropout (Vol %) at Room Temperature							BS&W (Vol %) After 24 h		
	5 min	30 min	1 h	2 h	3 h	4 h	24 h	FW	E	TW
HO Flow										
2000	15	20	22	23	23	-	23			
1000	20	20	22	25	25	25	25	6	6	11
500	25	30	30	30	30	30	30			
SRC-K1										
2000	10	15	20	20	-	-	23			
1000	15	22	25	25	25	-	25	6	8	8
500	W/O									
Flothin F-2										
2000	2	3	3	4	5	10	18			
1000	5	15	18	20	20	20	25	18	20	24
500	10	22	25	25	30	30	30			

COMMENTS ON EMULSION PREPARATION

	2000 ppm	1000 ppm	500 ppm
HO Flow	Good dispersion at 1 minute. Still large drops at 2 minutes	Same as 2000 ppm	Poor emulsion at 2 min. Very large oil globs.
SRC-K1	Good dispersion at 30-40 s. Complete dispersion at 1 minute.	Same as 2000 ppm	Good dispersion at 30-60 s, but large oil drops. Complete dispersion at 2 min. Converted into w/o emulsion at 6.5 min.
Flothin F-2	Complete dispersion at 30 s.	Good disperson at 30-50 s, complete at 1 min.	Good dispersion at 40-60 s, complete at 2 min.

Table 4. O/W Emulsification Characteristics of Texaco Lone Rock Wellhead Crude (B5-14-47-27 W3M)

Emulsifier (ppm)	Water Dropout (vol %) at Room Temperature							BS&W (Vol %) After 24 h		
	5 min	30 min	1 h	2 h	3 h	4 h	24 h	FW	E	TW
HO Flow										
2000	20	22	22	22	22	-	25			
1000	20	20	25	25	25	25	30	12	42	50
500	25	30	30	30	30	35	35			
SRC-K1										
2000	40	50	53	55	-	-	58			
1000	45	50	55	58	-	-	58	26	0	26
500	42	42	42	42	-	-	42			
Flothin F-2										
2000	8	20	20	22	25	25	30			
1000	20	25	30	33	35	35	35	46	2	48
500	30	30	35	35	37	37	37			

COMMENTS ON EMULSION PREPARATION

	2000 ppm	1000 ppm	500 ppm
HO Flow	Good dispersion at 1 min. Large oil drops. Beaker surprisingly oil-wet.	Same as 2000 ppm	Same as 2000 ppm, except very large oil drops.
SRC-K1	Complete dispersion at 10-15 s. Excellent o/w emulsion. Beaker water-wet. Broken-out water was oily.	Same as 2000 ppm, except dispersion complete at 20 s.	Dispersion complete at 30-40 s. Much less oily water.
Flothin F-2	Complete dispersion at 20-30 s. Very homogeneous emulsion; small droplets.	Complete dispersion between 30-60 s. Excellent emulsion at 60 s. Small droplets.	Complete dispersion at 30 s, homogeneous at 2 min. Very large oil drops.

Table 5. O/W Emulsion Viscosities of Texaco Frog Lake (McLaren) Wellhead Crude (8-13-57-2 W4M), Using Various Emulsifiers at Three Concentrations

RPM	Viscosity (mPa.s) at 22°C											
	HO Flow (ppm)			PD-57 (ppm)	SRC-K1 (ppm)			P-85 (ppm)	Flothin F-2 (ppm)			
	500	1000	2000	1000	500	1000	2000	1000	500	750	1000	2000
0.5	12730	13890	15620	6590	41100	7270	1360	1140	21990	3640	680	340
1.0	14520		15330		35040		965		19090	2960		170
2.5									14120			
5	11030	9660	7620				475					135
10												
20				2450		710	305	140		850	125	130
50												
100						95		60		45	65	

Table 6. O/W Emulsion Viscosities of Texaco Frog Lake (Waseca) Wellhead Crude (14-6-57-1 W4M) Using Selected Emulsifiers at Three Concentrations

RPM	Viscosity (mPa.s) at 22°C								
	HO Flow (ppm)			SRC-K1 (ppm)			Flothin F-2 (ppm)		
	500	1000	2000	500	1000	2000	500	1000	2000
0.5	13310	4320	4210	w/o	2840	680	17360	7390	115
1.0	11570	3580	3410		1820	450	8130	4550	115
2.5									
5	9210								
10		2930	2330		560	180	3130	450	170
20			1340						
50									
100					40	70		95	125

Table 7. O/W Emulsion Viscosities of Texaco Lone Rock Wellhead Crude (B5-14-47-27 W3M), Using Selected Emulsifiers at Three Concentrations

RPM	Viscosity (mPa.s) at 22°C								
	HO Flow (ppm)			SRC-K1 (ppm)			Flothin F-2 (ppm)		
	500	1000	2000	500	1000	2000	500	1000	2000
0.5	3640	2390	2610	2730	340	340	4550	1360	680
1.0	3180	1930	2100	2270	170	170	3980	680	340
2.5									
5									
10	1550	820	680	1420	30	30	1700	190	85
20									
50									
100	30	30	50	50	15	15	35	25	30

Table 8. Compatibility Studies - Demulsification Bottle Tests Following Emulsification of Actual Wellhead Crudes

Crude/Emulsifier /Demulsifier[a]	Water Dropout (Vol %)[b]							Final BS&W (Vol %)			
	15 min	30 min	1 h	2 h	3 h	4 h	5 h	FW	E	TW	S
Frog Lake 8-13											
Original Crude								0	0.3	0	0.4
888X	0	0	0	0	0	0	Trace	0	0.5	0	0.7
HO Flow/888X	0	0	Trace	2	3	3	3	2.0	0.4	2.4	0
SRC-K1/888X	0	0	0	Trace	1	2	2	3.0	1.0	3.6	0
Flothin F-2/888X	0	3	3	4	3	4	5	5.8	0	5.8	0
Frog Lake 14-6											
Original Crude								0	4.2	2.6	0.3
888X	0	0	0	0	0	0	0	0.4	3.0	3.2	0.4
HO Flow/888X	0	0	0	1	1	2	2	3.0	4.0	7.0	0
SRC-K1/888X	0	0	0	0	Trace	1	1	5.2	0	5.2	0
Flothin F-2/888X	0	2	2	3	4	5	10	5.0	2.0	7.0	0
Lone Rock B5-14											
Original Crude								36	20	54	0
888X	50	52	52	55	55	55	55	0.8	0	0.8	0
HO Flow/888X	45	45	47	47	50	50	50	6.0	1.0	7.0	0
SRC-K1/888X	15	22	22	27	22	22	22	1.0	0	1.0	0
Flothin F-2/888X	25	30	35	35	40	40	40	16	3.0	18	0

[a] Champion 888X demulsifier concentration: 300 ppm.

[b] Water dropout at 90°C for Frog Lake samples and at 80°C for Lone Rock samples.

Table 9. Compatibility Studies - O/W Emulsification of Artificial (High BS&W) Frog Lake Crudes

Crude[a]/ Emulsifier[b]	Water Dropout (Vol %) at Room Temperature		BS&W (Vol %) After 24 h		
	2 h	24 h	FW	E	TW
Frog Lake 8-13					
No o/w emulsification	0	0	19	12	27
HO Flow	35	38	22	2	25
SRC-K1	40	40	10	1	12
Flothin F-2	28	30	26	2	28
Frog Lake 14-6					
No o/w emulsification	0	0	16	24	29
HO Flow	30	30	15	15	26
SRC-K1	35	38	21	1	22
Flothin F-2	18	28	24	1	25

[a] Artificial crudes prepared by mixing 75 vol % wellhead crude and 25% vol % brine.

[b] Emulsifier concentration: 1000 ppm.

Table 10. Compatibility Studies--Demulsification Bottle Tests Following Emulsification of Artificial (High BS&W) Frog Lake Crudes

Crude[a]/Emulsifier /Demulsifier[b]	Water Dropout (Vol %) at 90°C							Final BS&W (Vol %)			
	15 min	30 min	1 h	2 h	3 h	4 h	5 h	FW	E	TW	S
Frog Lake 8-13											
No demulsifier	0	Trace	Trace	Trace	2	3	3	18	3	21	0
888X	2	5	5	18	18	18	18	3.2	0.8	4.0	0.7
HO Flow/888X	5	10	10	18	20	20	20	1.6	2.0	3.4	0
SRC-K1/888X	0	0	0	0	1	1	1	2.0	2.0	4.0	0
Flothin F-2/888X	2	5	8	15	15	20	20	4.0	0	4.2	0.6
Frog Lake 14-6											
No demulsifier	0	0	0	0	0	1	1	3.0	29	25	0
888X	0	0	Trace	3	20	22	22	4.0	4.0	8.0	0
HO Flow/888X	0	2	3	18	20	22	22	1.0	7.0	8.0	0
SRC-K1/888X	0	0	0	0	3	5	10	0	2.8	2.4	0
Flothin F-2/888X	1	3	5	18	20	20	20	4.7	0.5	5.2	0

[a] See Table 9, footnote a.

[b] Champion 888X demulsifier concentration: 300 ppm.

Table 11. Salinity Effect on O/W Emulsion Stabilities of Texaco Frog Lake Wellhead Crude (8-13-57-2 W4M)

Emulsifier (ppm)/ Brine (dilution)	Water Dropout (Vol %) at Room Temperature							Comments
	5 min	30 min	1 h	2 h	3 h	4 h	24 h	
HO Flow								
1000 ppm/1x dil	-	20	20	20	25	25	25	Good emulsion but large oil drops.
/10x dil	10	20	25	30	30	35	35	Same.
/100x dil	5	20	20	22	22	22	25	Same, but smaller drops.
500 ppm/1x dil	25	30	30	30	30	30	30	Very large oil globs. Marginal emulsion.
/10x dil	30	35	35	35	35	35	35	Same.
/100x dil	20	25	30	30	35	35	35	Same.
250 ppm/1x dil	w/o							Reversed after 6 min.
/10x dil	35	35	35	35	35	35	35	No emulsification.
/100x dil	25	25	25	25	30	30	30	Separate phases, some w/o emulsion at 3 min.
								All broken-out water relatively clean.
SRC-K1								
1000 ppm/1x dil	-	20	25	25	25	25	25	Good emulsion.
/10x dil	1	3	8	10	20	20	25	Good to excellent emuls.
/100x dil	1	3	10	18	20	20	22	Excellent emuls. with smaller drops.
500 ppm/1x dil	w/o							Reversed after 7 min.
/10x dil	25	30	35	35	35	35	35	Rapid dispersion but large drops.
/100x dil	25	30	30	30	30	30	35	Large globs, separate phases.
250 ppm/1x dil	w/o							Reversed after 2 min.
/10x dil	w/o							Reversed after 4 min.
/100x dil	w/o							Reversed after 7 min.

(continued)

Table 11. Salinity Effect of O/W Emulsion Stabilities of Texaco Frog Lake Wellhead Crude (8-13-57-2 W4M) - Continued

Emulsifier (ppm)/ Brine (dilution)	Water Dropout (Vol %) at Room Temperature							Comments
	5 min	30 min	1 h	2 h	3 h	4 h	24 h	
Flothin F-2								
1000 ppm/1x dil	-	3	3	8	15	15	18	Excellent emulsion, but oily water.
/10x dil	0	2	3	3	-	-	18	Same.
/100x dil	0	1	2	5	-	-	18	Same.
500 ppm/1x dil	2	15	20	20	-	-	22	Same.
/10x dil	0	1	3	5	-	-	20	Same.
/100x dil	0	2	5	10	-	-	20	Same.
250 ppm/1x dil	25	25	25	25	-	-	25	Fair emulsion with larger drops. Reversed at 4 min
/10x dil	1	10	20	20	-	-	22	Good emulsion but relatively large oil drops.
/100x dil	0	20	22	25	25	25	25	Marginal, large globs.

Table 12. Sand Dropout from Texaco Frog Lake (8-13-57-2 W4M) O/W Emulsions with a Sand Concentration of 15 Vol % Sand-in-Oil (10 Vol % Sand Overall)

Emulsifier	Water (W) and Sand (S) Dropout (Vol %) at Room Temperature				Comments[a]
	30 min	1 h	24 h	6 d	
Blank I (sand added only)					No sand dropout after 24 and 48 h at 30°C.
Blank II (mixed with brine without emulsifier)	w/o				Complete w/o emulsification after 5 min at high-speed stirring. After 24 h at 30°C, large pockets of clean sand and water were visible throughout oil phase. Top layer (10 vol %) was sand-free, also 10 vol % free water had settled out on bottom. After 48 h at 30°C, 12 vol % water and 2 vol % sand had settled out.
HO Flow 1000 ppm	w/o				Initially very large oil globs. After 2 min gradual reversal to w/o emulsion. Reversal complete at 6 min of low-speed stirring. Dropped-out sand was white. Water was clean.
SRC-K1 1500 ppm	w/o				Complete dispersion at 1 min, but large oil drops. Started to reverse to w/o at 6-7 min, complete at 10 min of low-speed stirring. Some oily water remained unemulsified.
1000 ppm	w/o				Complete dispersion at 1 min. Oily sand settled on bottom of beaker. Reversal to w/o complete after 15 min low-speed and 1 min high-speed stirring. Broken-out water very oily.
Flothin F-2 1000 ppm	W+S: 20	24	34		Nearly complete dispersion at 1 min. Good emulsion. Volume of dropped-out sand decreases as oil is released. Sand very clean after 24 h.
	S: 14	12	10		
500 ppm	w/o				Reversal to w/o complete after 10 min low-speed and 1 min high-speed stirring. Pockets of clean sand throughout w/o emulsion just after formation.

[a] Stirring: 15 min at low speed, followed by 1 min at very high speed.

Table 13. Sand Dropout from Texaco Frog Lake (8-13-57-2 W4M) O/W Emulsions with a Sand Concentration of 5 Vol % Sand-in-Oil (3.4 Vol % Sand Overall)

Emulsifier	Water (W) and Sand (S) Dropout (Vol %) at Room Temperature				Comments[a]
	30 min	1 h	24 h	6 d	
HO-Flow					
1000 ppm	W+S: w/o	w/o	30	32	Complete disperson at 1 min. Large drops increasing in size and at 6 min a partial reversal to w/o took place. Some clean sand settled in mixing beaker. Beaker fairly water-wet. At 24 h in cylinder, sand was very oily. Large oil drops were stuck to glass in water layer. Water was very oily.
	S:		6	0.8	
SRC-K1					
1000 ppm	W+S: -	23	30	32	Complete dispersion at 40-60 s. Good emulsion, but larger drops than with F-2. Drop size increased after 1 min high-speed stirring (reversal imminent?) No sand in mixing beaker. Beaker water-wet. At 24 h dropped-out sand was clean, the water oily.
	S: -	Trace	1	1	
Flothin F-2					
1000 ppm	W+S: -	12	16	26	Complete dispersion at 40-60 s. Good emulsion with small drops. No sand in beaker. Beaker water-wet. At 1 h a separated layer appeared at the bottom, consisting of sand, water and some oil. At 24 h dropped-out sand and broken-out water were oily.
	S: -	-	1	1	

[a] See footnote a, Table 12.

Table 14. Sand Dropout from Texaco Frog Lake (8-13-57-2 W4M) O/W Emulsions with a Sand Concentration of 2 Vol % Sand-in-Oil (1.4 Vol % Sand Overall)

Emulsifier	Water (W) and Sand (S) Dropout (Vol %) at Room Temperature				Comments[a]
	30 min	1 h	24 h	6 d	
HO-Flow 1000 ppm	W+S: -	28	28	29	Complete dispersion at 20-40 s. Large oil drops. Fair emulsion. Some fairly clean sand on bottom of beaker.
	S: -	0	Trace	≈0.6	
SRC-K1 1000 ppm	W+S: -	8	24	27	Complete dispersion at 30-50 s. Good emulsion, much smaller drops than HO Flow. No sand on bottom of beaker.
	S: -	0	Trace	≈0.6	
Flothin F-2 1000 ppm	W+S: -	0	10	19	Complete dispersion at 30-50 s. Excellent emulsion with smaller drops than SRC-K1. Broken-out water is very oily.
	S: -	0	Trace?	≈0.6	

[a] See footnote a, Table 12.

Table 15. Final BS&W Values of Texaco Frog Lake (8-13-57-2 W4M) Oil after Sand Settling Experiments

Emulsifier (1000 ppm)	BS&W (Vol %)*				
	FW	E	TW	S	
	5 Vol % Sand-in-Oil (3.4 Vol % Sand Overall)				
HO Flow	0	0.8	0.4	0.8	BS&W after 6 days
SRC-K1	0.4	0.4	1.0	0.4	id
Flothin F-2	16	10	18	0.8	id
	2 Vol % Sand-in-Oil (1.4 vol % Sand Overall)				
HO Flow	12	0	13	1.3	BS&W after 1 day
SRC-K1	15	0	15	1.6	id
Flothin F-2	30	0	31	0.6	id
HO Flow	9	9	8	2.0	BS&W after 6 days
SRC-K1	10	6	10	1.0	id
Flothin F-2	17	31	18	0.2	id

* Sampled at 200-mL mark in 500-mL graduated cylinder, i.e. about 3 cm above the oil/water interface.

Table 16. Sand Dropout from Texaco Frog Lake (14-6-57-1 W4M) O/W Emulsions with a Sand Concentration 15 Vol % Sand-in-Oil (10 Vol % Overall)

Emulsifier (1000 ppm)	Water (W) and Sand (S) Dropout (Vol %) at Room Temperature				Comments[a]
		30 min	1 h	24 h	
HO-Flow	w/o				Large blobs of oil formed at 30-60 s, signalling reversal to w/o. Reversal complete at 8 min. Some free water remained.
SRC-K1	w/o				Complete dispersion at 30-40 s. Marginal emulsion that started to reverse at 2 min. Reversal complete at 12 min. Beaker oil-wet.
Flothin F-2	W+S:	32	25	35	Complete dispersion at 30-40 s. Excellent emulsion, but high water content in suspended oil droplets (close to reversal?). Little sand on bottom of beaker. Separated sand and water very oily.
	S:	22	9	9	

[a] See footnote a, Table 12.

Table 17. Sand Dropout from Texaco Lone Rock (B5-14-47-27 W3M) O/W Emulsions with a Sand Concentration of 15 Vol % Sand-in-Oil (10 Vol % Overall)

Emulsifier (1000 ppm)	Water (W) and Sand (S) Dropout (Vol %) at Room Temperature			Comments[b]
	30 min	1 h	24 h	
HO-Flow	w/o			Large blobs of oil formed immediately and reversal to w/o followed. Much water did not emulsify and settled out. No sand on bottom of beaker. Beaker remained water-wet. At 24 h large pockets of entrained clean sand. At interface large oil globules.
SRC-K1	W+S: 64	-	64	Complete dispersion at 10-20 s. Good emulsion. Large amount of clean sand on bottom of beaker (more than with Flothin F-2).
	S: 4	-	6	
Flothin F-2	W+S: 48	-	52	Complete dispersion at 10-20 s. Good emulsion but large oil drops. Clean sand on bottom of beaker.
	S: 6	-	5	

[a] Sand used was from Frog Lake (14-6-57-1 W4M)

[b] See footnote a, Table 12.

FIG. 1 - CHEMICAL INJECTION SYSTEM